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Synthesis and Crystal Structure of Cobalt(III) Chelate with Tridentate Azomethine Ligand Containing a Benzimidazole Moiety

L. D. Popov[a, *], S. A. Borodkin[a], M. A. Kiskin[b], A. A. Pavlov[c], P. A. Knyazev[d], V. V. Chernyavina[a], and I. N. Shcherbakov[a]
[a] *Southern Federal University, Rostov-on-Don, Russia*
[b] *Kurnakov Institute of General and Inorganic Chemistry, Russian Academy of Sciences, Moscow, Russia*
[c] *Nesmeyanov Institute of Organoelement Compounds, Russian Academy of Sciences, Moscow, Russia*
[d] *Research Institute of Physical and Organic Chemistry, Southern Federal University, Rostov-on-Don, Russia*
**e-mail: ldpopov@mail.ru*

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Abstract—Azomethine HL was synthesized by the reaction of *o*-aminophenol with 2-acetylbenzimidazole and used to prepare the cobalt(III) complex $[CoL_2]_2(ClO_4)_2 \cdot 3H_2O$ (**I**). The structure of complex **I** was established by NMR spectroscopy in solution and by single crystal X-ray diffraction (CIF file CCDC no. 2051279). The cobalt ion in the +3 oxidation state has an octahedral environment. The crystals are monoclinic, space group $C2/c$, $a = 12.405(7)$, $b = 13.946(11)$, $c = 18.907(13)$ Å, $\alpha = 109.87(3)°$, $\beta = 94.534(12)°$, $\gamma = 104.564(19)°$, $V = 2928(3)$ Å^3, ρ(calcd.) = 1.521 g/cm^3, $Z = 2$. The complex is diamagnetic. Study of the electrochemical behavior of **I** in acetonitrile showed that it can be reduced to neutral and radical anion forms.

Keywords: azomethine, cobalt(III) complex, X-ray diffraction, NMR, cyclic voltammetry
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INTRODUCTION

Schiff bases (azomethines) occupy a top position for various fields of chemistry such as bioorganic [1, 2], medicinal [3, 4], supramolecular [5, 6], and coordination chemistry [7, 8], photochemistry [9–12], etc. Of particular interest are heterocyclic Schiff bases, as many heterocycles exhibit pharmacophoric properties and contain heteroatoms capable of forming stable bonds with metal ions.

In this study, 2-acetylbenzimidazole was chosen as the carbonyl component for azomethine synthesis. It is well known that benzimidazole derivatives possess diverse biological activities: antimicrobial [13], anticancer [14], antihistaminic [15], antituberculosis [16], antimalarial [17], antiviral [18], anti-HIV [19], and other. Furthermore, the benzimidazole moiety is a part of chromogenic chemosensors [20–22], OLED materials [23], catalysts [24], etc. Only few examples of 2-acetylbenzimidazole azomethines and complexes based on them were reported [25–30]; however, numerous examples of 2-aminobenzimidazole-based "reverse" azomethines and their complexes are known. It is known from the literature that structurally similar Co(II) complexes with a similar ligand, resulting from condensation of *o*-aminophenol with pyridine-2-aldehyde, exhibit spin crossover [31]. In addition, some cobalt(II) complexes show anomalous magnetic behavior, associated with retarded magnetic susceptibility relaxation [32, 33].

The purpose of this study was to investigate the complexing properties of 2-((1-(1*H*-benzo[d]imidazol-2-yl)ethylidene)amino)phenol (HL), formed as the condensation product of 2-acetylbenzimidazole with *o*-aminophenol, towards the cobalt(II) ion and to investigate the properties of the complex.

EXPERIMENTAL

The target compounds were synthesized using commercially available chemicals: 2-aminophenol, cobalt(II) perchlorate hexahydrate, methanol, toluene, and acetonitrile (Acros).

Synthesis of HL. A solution of 2-aminophenol (0.400 g, 3.80 mmol) in toluene (5 mL) was added to a hot solution of 2-acetylbenzimidazole (0.600 g, 3.80 mmol) [34] in toluene (10 mL). Formic acid (0.3 mL) was added to the resulting orange solution, and the mixture was refluxed for 4.5 h. The precipitate was collected on a filter, washed with hot toluene and diethyl ether, and recrystallized from ethanol. The yield of HL was 0.650 g (69%). T_m = 224–225°C.

For $C_{15}H_{13}N_3O$			
Anal. calcd., %	C, 71.73	H, 5.17	N, 16.72
Found, %	C, 71.40	H, 5.00	N, 16.40

IR (ν, cm^{-1}): 2200–3200 br.vs, 1634 s, 1586 s, 1484 m, 1449 vs, 1435 vs, 1345 m, 1320 s, 1292 s, 1223 s, 1149 w, 1103 w, 972 w, 827 w, 860 w, 757 vs, 742 m, 699 w, 608 w, 573 w. ^{1}H NMR (600 MHz; DMSO-d$_6$; δ, ppm): 12.97 (s, 1H, NH), 9.17 (s, 1H, OH), 7.75 (d, $^3J_{HH}$ = 8.0 Hz, 1H, Bz), 7.53 (d, $^3J_{HH}$ = 8.0 Hz, 1H, Bz), 7.30 (t, $^3J_{HH}$ = 8.0 Hz, 1H, Bz), 7.23 (t, $^3J_{HH}$ = 8.0 Hz, 1H, Bz), 6.98–7.02 (m, 1H, Ph), 6.92 (d, $^3J_{\underline{HH}}$ = 7.9 Hz, 1H, Ph), 6.85 (m, 2H, Ph), 2.33 (s, 3H, Me). ^{13}C{^{1}H} NMR (151 MHz; DMSO-d$_6$; δ, ppm): 160.09 (s, Bz), 151.22 (s, $\underline{C}$-Me), 147.08 (s, Ph), 143.28 (s, $\underline{C}$–NH), 136.93 (s, Ph), 134.80 (s, $\underline{C}$–N (Bz)), 125.30 (s, Ph), 124.14 (s, Bz), 121.95 (s, Bz), 120.80 (s, Ph), 119.99 (s, Bz), 119.28 (s, Ph), 116.03 (s, Ph), 112.12 (s, Bz), 16.96 (s, Me).

Synthesis of I. A hot solution of $Co(ClO_4)_2 \cdot 6H_2O$ (0.100 g, 0.145 mmol) in methanol (4 mL) was added to a hot solution of HL (0.150 g, 0.29 mmol) in methanol (5 mL). The resulting dark red solution was allowed to stand for 3 days. The precipitated crystalline solid was collected on a filter, washed with methanol, and dried in vacuum. The yield of **I** was 0.100 g (56%). According to X-ray diffraction data, the composition of the product was $[CoL_2]_2(ClO_4)_2 \cdot 3H_2O$.

IR (ν, cm^{-1}): 3236 br.w, 1588 m, 1568 m, 1537 m, 1322 s, 1256 vs, 1218 w, 1148 m, 1050–1150 br.vs, 1030 w, 994 w, 845 w, 746 s, 623 m, 564 m. ^{1}H NMR (300 MHz; DMSO-d$_6$; δ, ppm): 14.93 (br.s., 1H, NH), 8.16 (d, $^3J_{HH}$ = 7.4 Hz, 1H, Bz), 7.60 (d, $^3J_{HH}$ = 7.4 Hz, 1H, Bz), 7.33 (m, 1H, Bz), 7.26 (m, 1H, Bz), 7.05 (m, 2H, Ph), 6.71 (m, 1H, Ph), 6.60 (d, $^3J_{HH}$ = 8.0 Hz, 1H, Ph), 2.33 (s, 3H, Me).

Single crystal X-ray diffraction study of compound **I** was carried out on a Bruker Apex II diffractometer equipped with a CCD array detector (MoK_α, λ = 0.71073 Å, graphite monochromator) [35]; SADABS program package [36] was used to apply the semiempirical absorption corrections (T_{min}/T_{max} = 0.6320/0.7461). The structures were solved by direct methods and refined by full-matrix least squares in the anisotropic approximation for all non-hydrogen atoms. The hydrogen atoms at nitrogen and carbon atoms of the organic ligands and hydrogen atoms of water molecules were generated geometrically and refined in the riding model. The structure was refined with allowance for the disorder of the ClO_4 anion over two sites with occupancies of 0.733(6) and 0.267(6). The calculations were carried out by the SHELX program package [37] using OLEX2 [38]. Crystallographic parameters and structure refinement details for **I** at T = 150(2) K: $C_{60}H_{54}Cl_2Co_2N_{12}O_{15}$, M = 1371.91 g/mol, space group $P\bar{1}$, black prismatic crystal, 0.15 × 0.15 × 0.12 mm, a = 12.405(7), b = 13.946(11), c = 18.907(13) Å, α = 109.87(3)°, β = 94.534(12)°, γ = 104.564(19)°, V = 2928(3) Å^3, Z = 2, ρ(calcd.) = 1.521 g cm^{-3}, μ = 0.735 mm^{-1}, $1.90° \le \theta \le 28.28°$, sphere segment $-16 \le h \le 15$, $-18 \le k \le 18$, $-25 \le l \le 25$, 25075 measured reflections, 14383 unique reflections, 10026 reflections with $I > 2\sigma(I)$, R_{int} = 0.0321, GOOF = 1.021, R_1 ($I > 2\sigma(I)$) = 0.0593, wR_2 ($I > 2\sigma(I)$) = 0.1412, R_1 (all data) = 0.0950, wR_2 (all data) = 0.1603, $\Delta\rho_{min}/\Delta\rho_{max}$, e Å^{-3} = –0.664/1.228.

The structural data are deposited with the Cambridge Crystallographic Data Centre (CCDC no. 2051279; deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk/data_request/cif).

Elemental analysis was carried out on a Perkin-Elmer 240C instrument at the laboratory of microanalysis of the Southern Federal University.

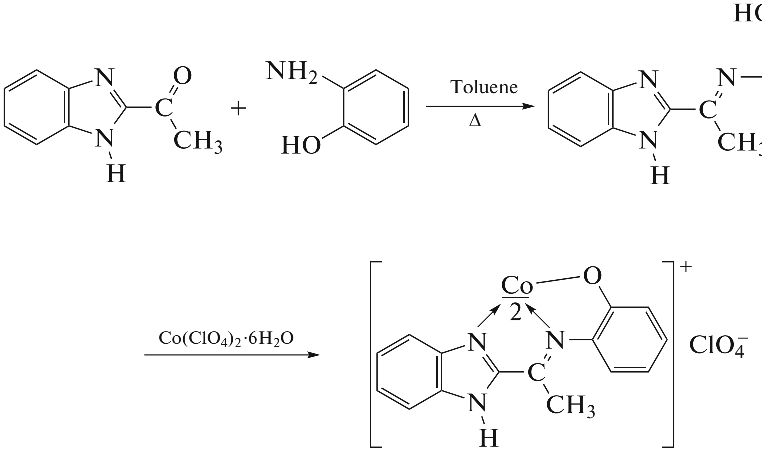
NMR spectra were recorded on Bruker Avance 300 and 600 spectrometers (operating at 300.13 and 600.22 MHz for ^{1}H). The chemical shifts (δ, ppm) were referred to the residual signal of the DMSO solvent (2.50 ppm for ^{1}H; 39.52 ppm for ^{13}C). IR spectra were measured on a Varian Scimitar 1000 FT-IR instrument in mineral oil in the 400–4000 cm^{-1} range. Electronic absorption spectra were obtained on a Varian Cary 5000 instrument in the 200–800 nm range. The diffuse reflectance spectra were recorded on a Varian Cary 5000 instrument with the Internal DRA 2500 attachment in the 400–1500 nm range.

The magnetic susceptibility of complex **I** was determined by the relative Faraday method at 295 K. The measurements were carried out at a magnetic field strength of 7.16 × 10^5 A/m. $Hg[Co(CNS)_4]$ was used as the calibration reference.

Cyclic voltammograms were recorded for acetonitrile solutions on a Elins-P45X potentiostat galvanostat in a three-electrode electrochemical cell. A glassy carbon working electrode, a platinum auxiliary electrode, and an Ag/Ag$^+$ (0.01 M $AgNO_3$) reference electrode were used. The potential sweep rate was v = 50 mV/s, and 0.1 M Bu_4NPF_6 served as the supporting electrolyte. Acetonitrile was purified before use. The low-molecular-weight organic impurities and traces of water were removed by fractional distillation. For this purpose, acetonitrile was stirred for 2 days in contact with calcium hydride (10 g/L), then decanted and subjected to fractional distillation from P_2O_5 (5 g/L). The product was heated at reflux over calcium hydride (5 g/L) for several hours, and then subjected to fractional distillation. The compound NBu_4PF_6 (Alfa Aesar) was preliminarily recrystallized from ethanol, dried, and calcined at 80°C for 4 h.

RESULTS AND DISCUSSION

The reaction of 2-acetylbenzimidazole with *o*-aminophenol on reflux in toluene gives rise to Schiff base (HL) in 69% yield (Scheme 1):



(I)

Scheme 1.

The IR spectrum of the obtained compound exhibits an intense band at 1634 cm^{-1} corresponding to C=N stretching vibrations of the azomethine moiety and a band at 1586 cm^{-1}, which can be assigned to vibrations of the endocyclic C=N and C=C bonds. In addition, the broad asymmetric intense band, due to formation of hydrogen bonds involving O–H and N–H groups, is observed in the 2200–3200 cm^{-1} range.

The characteristics of signals in the ^1H and $^{13}C\{^1H\}$ NMR spectra of HL (chemical shifts, integrated intensities, and multiplicities) are in full agreement with the presumed structure. Complete signal assignment was carried out using 2D NMR procedures: ^1H–^1H COSY, ^1H–^{13}C HMQC, and ^1H–^{13}C HMBC.

The electronic absorption spectra of HL in ethanol exhibit an absorption band with a maximum at λ = 300 nm (logε = 4.00) and a shoulder at λ_{max} = 358 nm (log ε = 3.36), which is attributable to the $\pi \rightarrow \pi^*$ transitions in the benzene and benzimidazole moieties, respectively (Fig. 1).

The reaction of $Co(ClO_4)_2{\cdot}6H_2O$ with HL in methanol (Scheme 1) gives complex **I**. In comparison with the spectrum of the ligand, the IR spectrum of complex **I** has only one new absorption band at 3236 cm^{-1}, corresponding to N–H stretching vibrations of the benzimidazole moiety. Simultaneously, the C=N vibration frequencies decrease by 20–50 cm^{-1}, indicating that the nitrogen atoms of these groups are involved in the coordination to the cobalt ion. The highly intense broad band at 1050–1150 cm^{-1} attests to the presence of the perchlorate ion in complex **I**.

Three absorption bands can be distinguished in the electronic absorption spectra of a solution of **I** in eth-

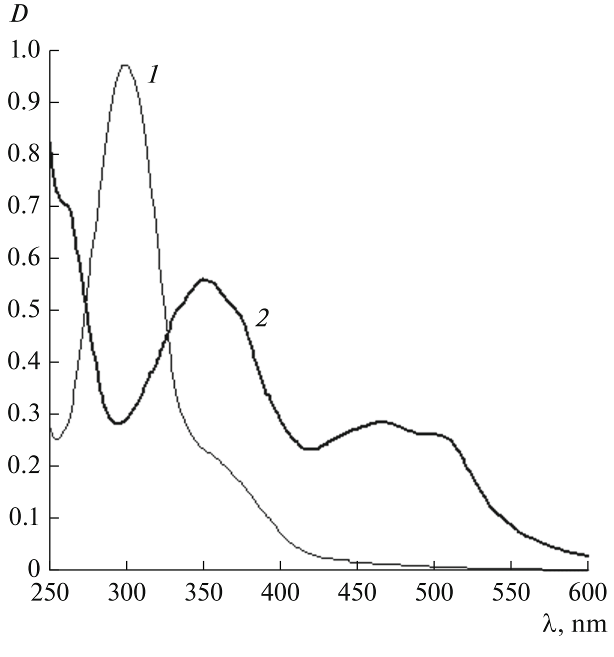


Fig. 1. Electronic absorption spectra of (*1*) HL ($c = 1 \times 10^{-4}$ M) and (*2*) **I** ($c = 2 \times 10^{-5}$ M) in ethanol.

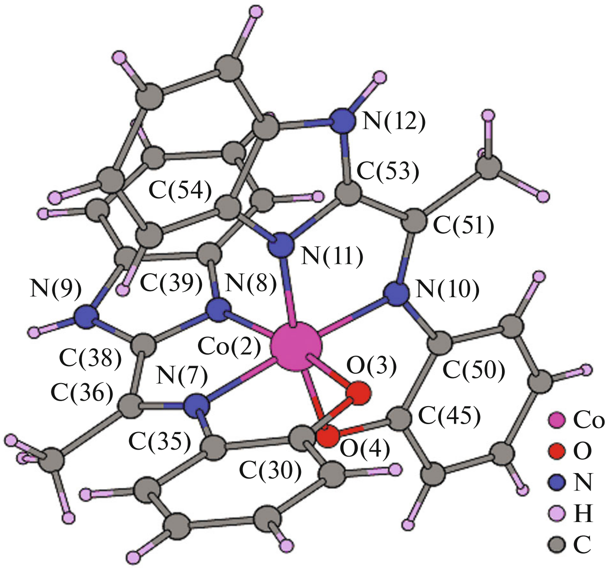


Fig. 2. Structure of the $[CoL_2]^+$ cation in **I**.

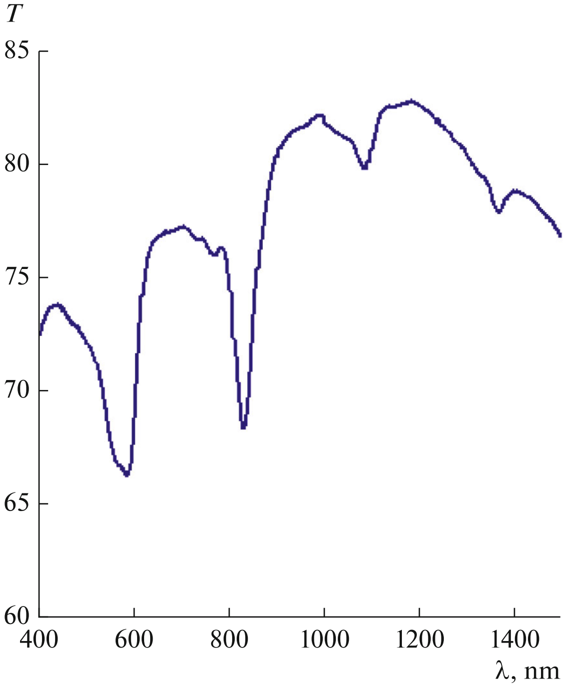


Fig. 3. Diffuse reflectance spectrum of **I**.

anol (Fig. 1). In comparison with the spectrum of HL, it can be noted that both $\pi \rightarrow \pi^*$ transitions are red-shifted by 50–100 nm. The third absorption band present in the spectrum at $\lambda_{max} = 505$ nm is probably caused by the M → L charge transfer.

The structure of complex **I** was determined on the basis of X-ray diffraction data. Compound **I** crystallizes in the triclinic space group $P\overline{1}$ as a water solvate. The independent part of the unit cell consists of two $[CoL_2]^+$ cations of a similar structure, two ClO_4^- anions, and three water molecules ($[CoL_2]_2(ClO_4)_2 \cdot 3H_2O$). Each cobalt atom coordinates two N,N,O-tridentate chelating ligands to give fused five-membered metallacycles (Co(1)–O, 1.881(2), 1.903(3); Co(1)–N, 1.917(3)–1.944(3) Å; XCo(1)X, 82.68(12)°–100.94(12)°; Co(2)–O, 1.878(3), 1.888(3); Co(2)–N, 1.900(3)–1.935(3) Å; XCo(2)X, 82.66(13)°–99.91(12)°) (Fig. 2). The geometry of the CoN_4O_2 polyhedra corresponds to a distorted octahedron. The metal–donor atom bond lengths in both $[CoL_2]^+$ cations are comparable and are characteristic of low-spin cobalt(III) ions. The aromatic moieties of the ligands occur in one plane; the deviation from the averaged plane does not exceed 0.25 Å, while the angles between the ligand planes in the $[CoL_2]^+$ cations are close to right angle, 86.04(6)° and 88.95(6)°. Intermolecular hydrogen bonds between ligand N–H groups and ligand oxygen atoms of the neighboring cation, outer-sphere ClO_4 anions, and water molecules are present in the crystals. Water molecules are hydrogen-bonded to the oxygen atoms of the ligand and to the

Table 1. Interactions D–H...A in the crystal of **I**

H-bond	D–H, Å	H...A, Å	D...A, Å	D–H...A, deg	Symmetry codes of A atom
N(3)–H(3)...O(4S)	0.88	2.08	2.899	154.3	$-x + 2, -y + 2, -z + 2$
N(3)–H(3)...O(4SA)	0.88	2.11	2.972	166.4	$-x + 2, -y + 2, -z + 2$
N(12)–H(12A)...O(1)	0.88	1.90	2.648	141.7	
N(6)–H(6)...O(1*w*)	0.88	1.92	2.766	160.1	
N(6)–H(6)...O(2SA)	0.88	2.19	2.852	131.4	
N(9)–H(9)...O(2*w*)	0.88	1.86	2.735	175.9	$-x, -y, -z + 1$
O(2*w*)–H(2*w*A)...O(8S)	0.85	2.21	3.008	157.6	
O(2*w*)–H(2*w*B)...O(4)	0.86	1.95	2.777	153.1	

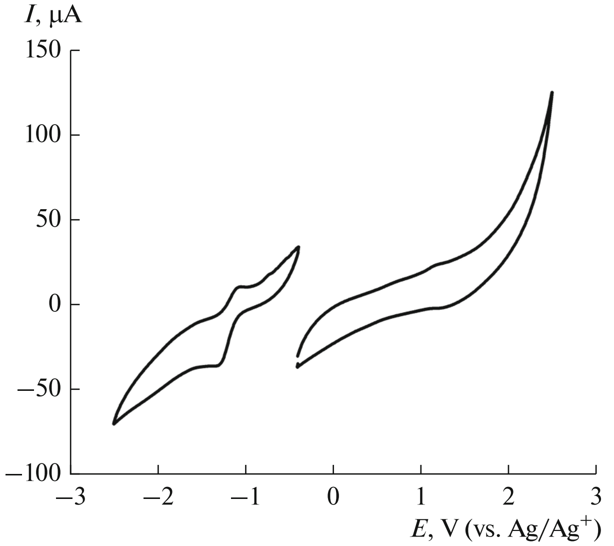


Fig. 4. Cyclic voltammograms for (on the right) oxidation and (on the left) reduction of a solution of HL (MeCN, $c = 3$ mmol/L, 0.1 M Bu_4NPF_6, $v = 50$ mV/s).

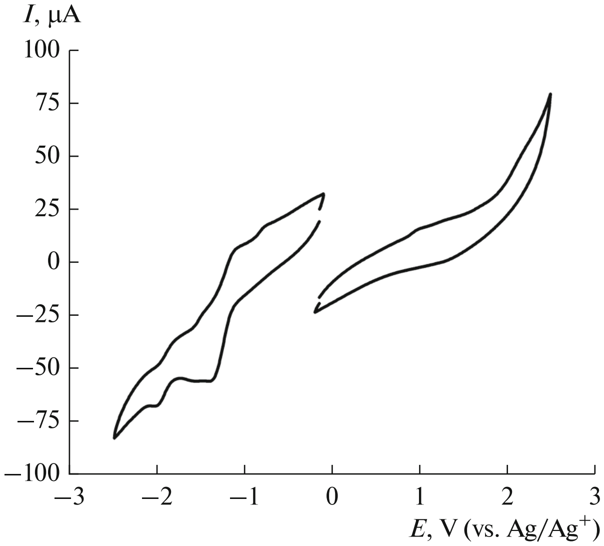


Fig. 5. Cyclic voltammograms for (on the right) oxidation and (on the left) reduction of a solution of complex **I** (MeCN, $c = 3$ mmol/L, 0.1 M Bu_4NPF_6, $v = 50$ mV/s).

ClO_4^- anions. Selected parameters of the hydrogen bonds are summarized in Table 1.

Study of the magnetic susceptibility of complex **I** in the solid state showed that the complex is diamagnetic. This is characteristic of compounds of low-spin cobalt(III) ions. The octahedral environment of cobalt in complex **I** is confirmed by the presence of two absorption bands in the diffuse reflectance spectra at 820 and 1080 nm, corresponding to $^1A_{1g} \to {}^1T_{1g}$ and $^1A_{1g} \to {}^1T_{2g}$ transitions, respectively (Fig. 3), which are also typical of Co^{3+} ion located in a strong ligand field [39].

The ^1H NMR chemical shifts of complex **I** differ from those of the free ligand HL. The most pronounced changes are observed for the protons located most closely to the sites of formation of coordination bonds with the cobalt ion, which confirms the complex formation in solution. The integrated intensity and multiplicity of the signals are also in line with the structure of complex **I**. In addition, the relatively small signal width and invariability of chemical shifts with changes in temperature attest to the diamagnetic nature of complex **I** in solution.

The most probable mechanisms of formation of Co(III) complex include the oxidation of Co^{2+} ion either with the ligand or with air oxygen present in the solvent. To verify this assumption, synthesis of the complex was carried out under inert atmosphere (argon) and in the presence of a reducing agent (ascorbic acid). The complex isolated upon this reaction was fully structurally identical to **I**, which was confirmed by X-ray diffraction data. Thus, the oxidation of the cobalt ion is evidently related to the nature of the ligand system.

Since the synthesis of complex **I** is accompanied by oxidation of cobalt to the trivalent state, it would be reasonable to study the electrochemical properties of **I**. The electrochemical behaviors of HL and complex **I** were studied by cyclic voltammetry (CV) in acetonitrile. Free ligand is reduced via one-electron quasi-reversible step at the potential $E_{1/2}^{\text{red}} = -1.16$ V to give the corresponding radical anion. Note that in the potential range down to −2.50 V, no additional redox transitions, indicating the formation of the dianionic form, were observed (Scheme 2).

$$\text{HL} \underset{-e}{\overset{+e}{\rightleftarrows}} [\text{HL}]^{-\cdot} \not\longrightarrow [\text{HL}]^{2-}$$

Scheme 2.

In the anodic region, no clearly defined oxidation peaks are present for HL (Fig. 4).

In the case of the cobalt complex, the CV curve (Fig. 5) exhibits two cathodic peaks at the potential $E_{1/2}^{\text{red1}} = -1.27$ V and $E_{1/2}^{\text{red2}} = -1.92$ V (versus Ag/Ag^+), corresponding to the initial reduction to the neutral form and then to the radical anion, due to participation of one of the ligands in the electrode process (Scheme 3).

$$[\text{CoL}_2]^+ \underset{-e}{\overset{+e}{\rightleftarrows}} [\text{CoL}_2] \underset{-e}{\overset{+e}{\rightleftarrows}} [\text{CoL}_2]^{-\cdot}$$

Scheme 3.

The potential of the first cathodic peak of complex **I** correlates with the reduction potential of a cobalt chelate with a similar environment [40]. The shape of the CV curves points to quasi-reversibility of the reduction reactions (Scheme 3) taking place on the electrodes. In the positive potential region, redox processes are poorly distinguishable.

Thus, it was shown that the obtained new Schiff base, representing a tridentate azomethine system, behaves as an oxidant towards Co^{2+} ions and, therefore, the cobalt complex with ligand of this type cannot exhibit spin crossover. The geometry of the CoN_4O_2 polyhedra in complex **I** corresponds to a distorted octahedron. In the crystal, there are intermolecular hydrogen bonds between the N–H groups of the ligand and oxygen atoms of the ligand belonging to the neighboring cations, outer-sphere ClO_4^- anions, and water molecules.

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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